

GE Corporate Research and Development

Research and Development Center General Electric Company PO. Box 8, Schenectady, NY 12301 518 3871. 11,12,13 O...

SDMS DocID 60175

Building K1-3B29 January 23, 1989

Dr. James L. Lake EPA, Environmental Research Laboratory Narragansett, RI 02882

Dear Jim:

I have just been informed by Paul Galvani, counsel for Aerovox, that I should turn over to EPA several classes of documents relating to the analytical work that we did on Acushnet River sediments back in 1986, and that you could serve as the official EPA recipient of this material. The documentation requested consisted of essentially all available information relating to:

- 1. Sampling procedures and chain-of-custody proofs
- 2. Analytical protocols used
- 3. All analytical data
- 4. Computer tapes of data.

As you may recall, on November 21, 1988 I sent you a voluminous data package, which included both computer print-outs and gas chromatograms of all 24 samples that we analysed, along with a map showing the points of origin of the specimens analysed, GM-MS data, and tables that compiled and summarized our analytical results; in short, all of the available information pertinent to requests (3) and (4). In this letter I include all the data I have regarding requests (1) and (2). Regarding the latter, I could further point out that our procedures have also been described in two of our published papers (Northeast. Environ. Sci., 3:167-179, 1984; Environ. Toxicol. Chem., 6:579-593, 1987). At any event, you are now in possession of all the requested documentation, which I suppose you will have to keep on file until one of your legal colleagues asks for it.

To move from legalism to science, we have been continuing to examine the chromatograms of both biota and sediments from various sites, and continuing to turn up new PCB alteration patterns now and then. At the moment, it looks as though the Pattern H and H' dechlorination, which we found in the upper Achushnet Estuary, may also be occurring at a great many (though not all) sites in the Hudson Estuary, all the way from Troy to the Battery. Fragmentary data suggests that Newark Bay may be a different story, however, as may also be Long Island Sound, and I'd still be interested in any of your samples or chromatograms that indicated what was going on off the rest of the New England Coast.

We have also been able to duplicate a dechlorination that looks very much like Pattern H' in culture, using an anaerobic microbial growth procedure

slightly different from the one John Quensen used to bring up a dechlorination similar to upper Hudson sediment Pattern C. I suspect that someone from our group may be talking about this at our semi-annual Region 1 seminar in Boston (actually, the Cambridge Marriott this time, I think) on February 9. Unfortunately, I won't be able to be there myself, owing to long-standing arrangements for cross-country skiing in Glacier National Park that week, but my colleagues will be able to convey messages or samples.

Sincerely yours,

John F. Brown, Jr.

Manager-Health Research

Biological Sciences Laboratory

John 2 Brom. J.

JFB/j

Encl.

cc: HL Finkbeiner

PB Galvani

ACUSHNET SEDIMENT SAMPLING FOR GE ANALYSES Documentation Package

<u>History of GE work on Acushnet Estuary Sediments</u> (i.e. Site and Sample Selection)

- 1. May, 1986. Examined collection of old gas chromatograms run by Versar in 1982-83. Found evidence of widespread but limited dechlorination.
- May 26, 1986, Telephoned finding to Stu Richardson of Aerovox. Indicated need for new samples for high resolution capillary GC to characterize transformation. He suggested I talk to Paul Galvani, his attorney.
- 3. I agreed to undertake capillary GC analyses on 24 specimens--taken at two levels (2-3" and 6-7") from each of 12 sites -- 6 on each side of estuary. Samples were to be collected by GHR Analytical by walking out on mud flats at low tide, digging a hole, and taking sediments at designated depths.
- 4. June 9, 1986 GHR sent GE samples from 26 sites (received 6-10-86). JF Brown selected 12 pairs for detailed PCB analysis and submitted them to RE Wagner (analyst) for analysis for procedure in use in laboratory.

Documentation in Package

- a. Chain-of-custody forms for samples.
- b. Maps showing GHR sampling locations.
- c. JF Brown's record of sample examination and selection for analysis.
- d. GHR report on oil and grease analyses on sediments.
- e. GHR certification.

John F. Brown, Jr. (1-20-89)

JFB/j

CHAIN OF CUSTODY RECORD

GHR Analytical Inc. 26 Main St., Lakeville, MA

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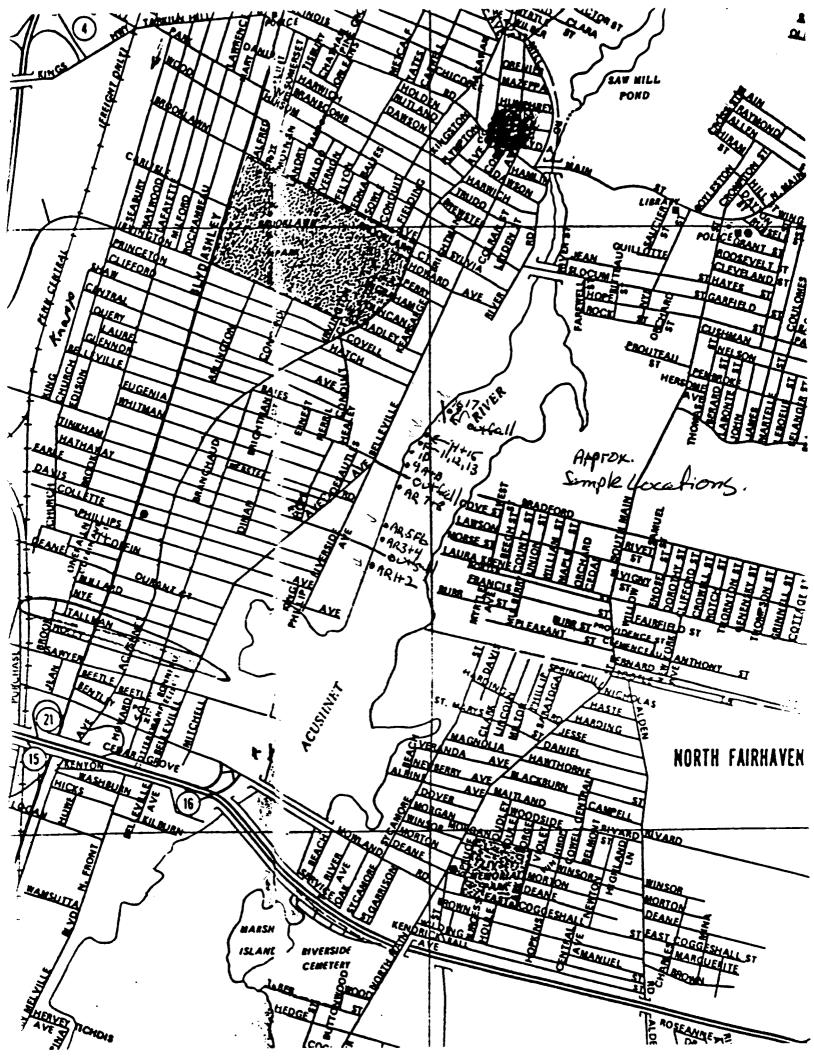
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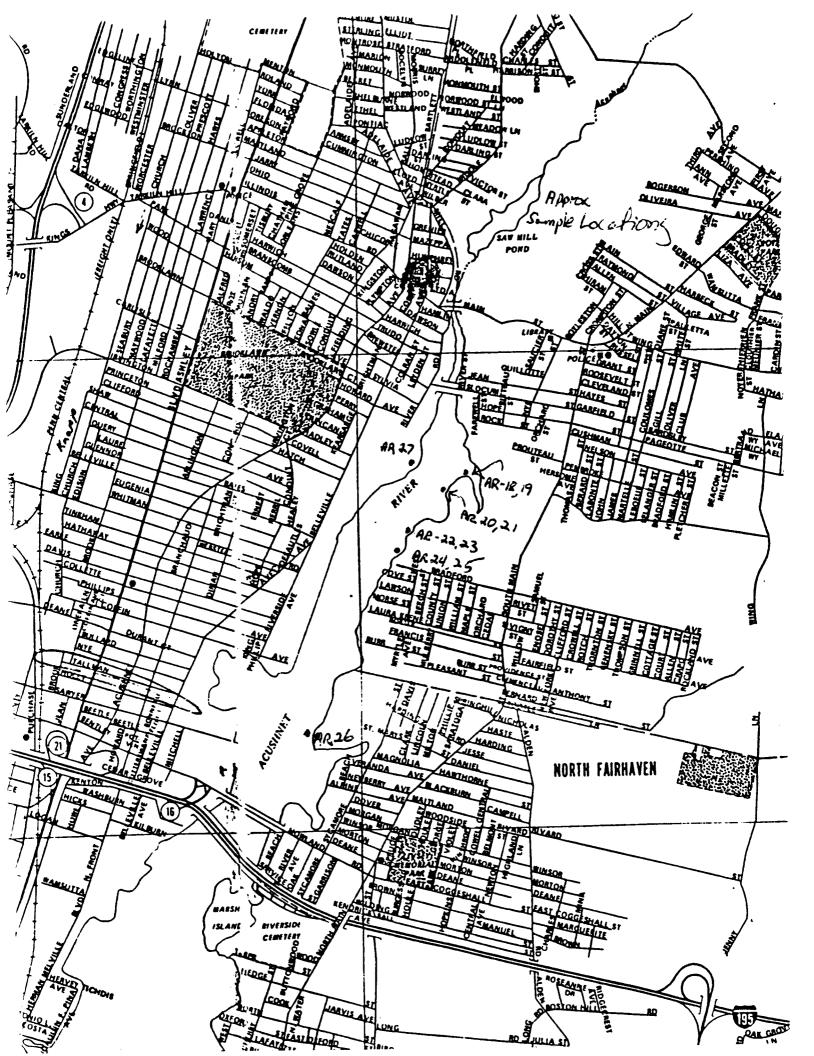
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* Basis for sample selection on 6/10/86:

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 despite my request for only 12 pairs of samples.
- · In salecting sites for study, rejected a few where sample all stones, grand, coarse sand - anticipated a low PCB contamt.
- even specing of sites, and veriety of sediment types.

GHR ANALYTICAL, INC. 26 MAIN STREET LAKEVILLE, MA 02347 (617)947-5077

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RESULTS OF SEDIMENT ANALYSIS

Acushnet River Estuary
Date Collected: June 6, 1986

Client: General Electric

Project: Oil & Grease Analyses

Job No.: 29-272

Date: June 17, 1986

Sample_Location	Depth	GHR_Lab_ID	Oil & Grease mg/kg_(dry_weight_basis)
AR-1A	3"	56114	1,040
AR-1B	7"	56115	780
AR-2A	3"	56116	1,570
AR-2B	7"	56117	2,050
AR-3A	3"	56118	1,700
AR-3B	7"	56119	2,430
AR-4A	3"	56120	2,630
AR-4B	7"	56121	980
AR-5A	3"	56122	12,800
AR-5B	7"	56123	34,500
AR-6A	3"	56124	78, 000
AR-6B	7"	56125	172,000
AR-7A	3"	56126	19, 300
AR-7B	7"	56127	6, 100
AR-8A	3"	56128	3,590
AR-8B	7"	56129	1,220
AR-9A	3"	56130	26, 7ଉଡ
AR-9B	7"	56131	22, 900
AR-10A	3"	56132	1,260
AR-1@B	7"	56133	421
AR-11A	3"	56134	926 Lu AM
AR-11B	7"	56135	261

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RESULTS OF SEDIMENT ANALYSIS

Acushnet River Estuary
Date Collected: June 6, 1986

Client: General Electric

Project: Oil & Grease Analyses

Job No: 29-272

Date: June 17, 1986

Sample Location	Depth	<u>GHR_Lab_ID</u>	Oil & Grease <u>mg/kg_(dry_weight_basis)</u>
AR-12A	3"	56136	8,730
AR-12B	7"	56137	6,070
AR-13A	3"	56138	10,900
AR-13B	7"	56139	4,840
AR-14A	3"	56140	3,840
AR-14B	7"	56141	3, 390
AR-15A	3"	56142	2,680
AR-15B	7"	56143	2,990
AR-16A	3"	56144	16, 300
AR-16B	7"	56145	21,600
AR-17A	3"	56146	46, 300
AR-17B	7"	56147	40, 300

GHR ANALYTICAL, INC. 26 MAIN STREET LAKEVILLE, MA 02347 (617) 947-5077

RESULTS OF SEDIMENT ANALYSIS

Acushnet River Estuary
Date Collected: June 10, 1986

Client: General Electric

Project: Oil & Grease Analyses

Job No.: 29-297

Date: June 17, 1986

Sample Location	Depth	GHR_Lab_ID:	Oil & Grease mg/kg (dry weight basis)
AR-18A	3"	56242	20, 700
AR-18B	7"	56243	7, 040
AR-19A	3"	56244	20,000
AR-19B	7"	56245	28, 400
AR-20A	3"	56246	5, 290
AR-20B	7"	56247	306
AR-21A	3"	56248	11, 100
AR-21B	7"	56249	1,440
AR-22A	3"	56250	5, 390
AR-22B	7"	56251	8,110
AR-23A	3"	56252	1,700
AR-23B	7"	56253	794
AR-24A	3"	56256	(150
AR-24B	7"	56257	(150
AR-25A	3"	56254	968
AR-25B	7"	56255	484
AR-26A	3"	56260	(440
AR-26B	7"	56261	(370

GHR ANALYTICAL INC. 26 MAIN STREET LAKEVILLE, MA 02347 (617) 947-5077

The information contained in this report is to the best of my knowledge, accurate and complete.

Leanne E.S. Cobb, Laboratory Manager

GHR Analytical Inc.

GE Corporate Research and Development Center

Material Characterization and Engineering Support Operation

Separations and Molecular Weights Lab

PCB MANUAL

Table of Contents

Part 1 - Organization Chart

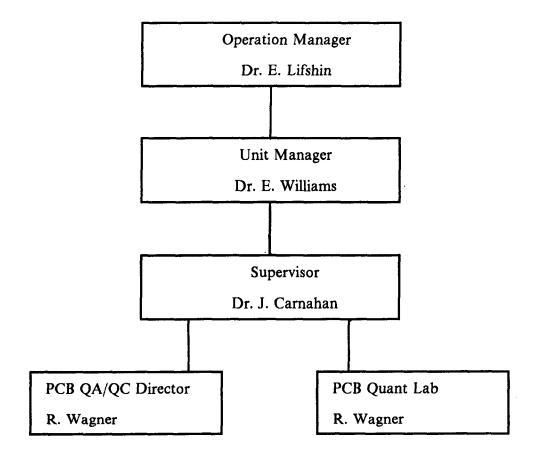
Part 2 - QA/QC Protocol

Part 3- References

Part 4 - PCB Protocol Manual

Part 1 - Organization Chart

Organization Chart



Part 2 - QA/QC Protocol

Quality Assurance and Quality Control

Sample Chain Of Custody

Samples entering the Lab are directed to the chemist who is in charge of the particular analysis requested. All samples are recorded into a sample notebook and assigned a sample ID number. A sample information sheet corresponding to the sample notebook number is filled in with all the necessary sample information. A peel off label with the sample ID number is attached to the vial containing the sample and the sample is then stored under appropriate conditions.

Calibration

Before every group of PCB analyses a Standard Aroclor is analyzed on the GC to check the calibration accuracy. If we find the Aroclor standard varies more than \pm 10% from true value, the instrument is calibrated and a standard is once again checked before samples are analyzed.

Aroclor Standard Reference

Aroclor standards were obtained from the CR&D stockroom, supplied by Monsanto Co. for GE's research programs. These bulk neat standards have been checked against NBS certified Aroclor in Oil standards as well as the Water Pollution QC samples supplied by EMSL, Quality Assurance Branch, USEPA, Cinn., OH. Our in-house standards agree very well with the two certified check samples.

Sample Precision and Accuracy

Accuracy of the instrumentation is tested by running standards to check on calibration quality. Testing extraction and clean up procedures were done when they were being developed to insure no sample loss at each step. If any changes occur in sampling methodology, it is checked with processing standards to insure sample integrity is being maintained. Precision of the analytical instrumentation is tested by occasionally running samples twice. Duplicate agreement must be maintained to \pm 10%. Testing procedures were initially examined using standardized materials and run separately to insure reproducibility and therefore good precision.

Rlanks

Solvent wash vials are added at the end of a group of samples. This allows us to clean the sampling system and also to check for baseline deviations or matrix effects and spurious peaks.

Data Reduction and Reporting

Refer to the PCB protocol manual for examples of GC data reporting. Data is collected by digitizing an analog detector signal and electronically integrating the area under a peak. Most instrument companies have available integrators that match their GC instrumentation and all GC integration is done automatically. Final result calculations can be programmed into the integrator and directly report sample amounts in the appropriate units (ug/g, ug/ml, ppm, etc.)

Instrument Conditions

See PCB Protocol Manual

Sample Preparation

See PCB Protocol Manual

Instrument Maintenance

The GC is maintained usually by the chemist who is utilizing the instrument, since he is most familiar with its status. Gas tanks are inspected and changed as needed. Injection port septa are changed after 50-60 injections. Noise level of the GC detector is monitored and the detector replaced once it become excessive. GC columns are replaced as needed, either if they become badly contaminated or have reached their lifetime and no longer perform the separation properly.

Part 3 - References

References

The following references have been used to develop the PCB methodology used at GE CR&D.

"Handbook of Quality Assurance for the Analytical Chemistry Laboratory", J Dux, Van Nostrad Reinhold publisher, 1986

"The Determination of PCB in Transformer Fluid and Waste Oils", TA Bellar & JJ Litchenberg, USEPA, 1981

"Quantitative PCB Standards for Electron Capture Gas Chromatography", RG Webb & AC McCall, Journal of Chromatographic Science, Vol 11, pp 366-373, 1973

"Recommended Analytical Requirements of PCB Data Generated On Site During Non-Thermal PCB Destruction Tests", USEPA, March 19, 1986

"The Federal Register, Title 40 CFR, Intermin Guidelines for Establishing Testing Procedures", USEPA, 1979

"Analytical Chemistry of PCBs", MD Erickson, Butterworth Publishers, 1986

"The Chemistry of PCB's", O Hutzinger, S Safe, & V Zitko, CRC Press, 1974

Part 4 - PCB Protocol Manual

INTRODUCTION

This protocol details the analysis of polychlorinated biphenyls (PCB's). The analytical procedure can be divided into three sections:

- I. Sample collection, preparation, and extraction: this section describes how to collect samples, preliminary sample preparation, and extraction of PCB's from the following sample matrices— soil and sediments, water, fish and other biological samples, oil, and air.
- II. Sample cleanup: this section describes the steps necessary to remove any materials co-extracted with the PCB's that may interfere with the subsequent analysis.
- III. Analysis: this section describes both the qualitative and quantitative analysis of PCB's by either packed or capillary column gas chromatography.

In addition, there are general guidelines which are followed for all sample types. These involve the types of materials which may contact the samples, how to clean glassware, and the quality of chemicals and solvents which must be used in the procedure.

1.0 GENERAL PROCEDURES

1.1 Glassware

To prevent interference in the analysis of trace substances, all glassware must be washed thoroughly prior to use. Specialty glassware is washed with soap and water and thoroughly rinsed with water. Next, it is rinsed with acetone, followed by hexane. Stockroom glassware is visually inspected for cleanliness. As an additional precaution, each piece of clean glassware is rinsed with hexane immediately prior to use.

1.2 Chemicals

All chemicals and solvents must be ACS reagent grade or better.

1.3 Other materials

No equipment containing plastic may be used. Plastic materials (such as Tygon tubing) give off phthalate esters, which interfere with PCB analysis and are difficult to remove. Only materials made from glass, teflon, or stainless steel may contact the sample during the procedure. All utensils must be pre-rinsed with hexane. All containers must be glass and have a teflon or foil-lined lid. All tubing used during such steps as solvent evaporation or vacuum drying must be either teflon or stainless steel.

2.0 SAMPLE COLLECTION, PREPARATION, AND EXTRACTION

2.1 Soil and Sediments

2.1.1 Sample collection

Soil and sediment should be collected in a wide-mouthed one quart screw-capped glass jar. The jar should be filled nearly to the top, using a pre-rinsed stainless steel utensil. If the sample is sediment, it should be "topped off" with sample water. The sample should be extracted as soon as possible; until extraction it should be stored at 4 degrees C.

2.1.2 Sample preparation

The whole sample(if practical) or a representative portion is spread out in a pyrex dish and allowed to air-dry for 24 hours in a hood. The sample is then sieved through a wire mesh screen to remove twigs and stones, and thoroughly mixed. An aliquot is placed in a small tared metal pan and dried overnight at 105 degrees C in a vacuum dessicator oven for dry weight determination. A separate aliquot(commonly 20-30g) is taken for extraction.

2.1.3 Extraction

Soil and sediment samples are extracted in a soxhlet apparatus. The standard-sized round-bottomed flask used is 250 ml and the standard thimble is a Whatman cellulose thimble with the dimensions 33 mm I.D. by 94 mm external length. The complete apparatus, including thimble and glass wool plug, is pre-extracted with hexane for several cycles(approximately 1 hr), allowed

to cool and the thimble allowed to air-dry. The sample is weighed into the tared thimble and the glass wool plug is inserted on top of the sample. A pre-extracted boiling stone is added to the boiling flask. The soil is then extracted overnight with 175 ml of a 1:1 mixture of hexane:acetone, and the apparatus is allowed to cool. All solvent is drained from the soxhlet into the round-bottomed flask.

2.2 Aqueous samples

2.2.1 Sample collection

Water samples are collected and stored in a manner similar to that for the collection of soil and sediment, as explained in Section 2.1.1.

2.2.2 Sample preparation

The aqueous sample is thoroughly shaken to evenly suspend any solids. If it is desirable to remove solids, the water is filtered through pre-extracted filter paper. Since aqueous solubility of PCB's is extremely low, a minimum water sample of 100 ml is desirable.

2.2.3 Extraction

Pour a known volume of aqueous sample into an appropriately sized glass bottle. Rinse the graduated cylinder used to measure the sample volume with 20 ml of a 15% methylene chloride in hexane solution(per 100 ml of sample) and add it to the glass bottle containing the sample. Cap the bottle, manually shake it and vent the vapor pressure as necessary, and place it on a

mechanical shaker for 20 minutes.

Allow the layers to separate. If an emulsion forms, add some sodium chloride and mix by inverting the bottle 2-3 times. If this does not separate the layers, the sample may be sonicated for several minutes. Withdraw the upper organic layer with a 10 ml pipet and transfer it to a 100 ml glass bottle. Re-extract the aqueous layer 2 more times as described; combine the organic layers.

2.3 Biological samples

2.3.1 Sample collection

The fish or other samples are collected, individually wrapped in hexane-rinsed aluminum foil, and frozen until subsections can be taken.

2.3.2 Sample preparation

Prior to subsectioning each sample is thawed The sample is subsectioned as desired and a weighed. 20 grams wet weight is used for the minimum of analysis(if possible). The sample is scissor-minced as finely as possible and suspended in approximately 50 ml of water in a 150 ml beaker. The sample is homogenized with a Brinkman Instruments Polytron. The Polytron probe is pre-rinsed by submerging it in hexane and turning the Polytron on. Homogenize at a low to medium setting(be careful not to splash) until the sample is a uniform consistency. The probe must be frequently of connective tissue buildup during this cleared process for efficient homogenization. To accomplish

this, turn off the instrument and use forceps to remove lumps of connective tissue from the probe tip. These may be minced and returned to the sample beaker.

The homogenate is next ground with anhydrous magnesium sulfate to further disintigrate the sample and to combine with the water that is present. accomplished in the following manner: transfer the homogenate to a glass mortar, using small quantities of water to rinse the beaker if necessary. sulfate in the ratio of 3.2 grams of magnesium magnesium sulfate per gram of wet tissue weight. Add the magnesium sulfate, in small portions, to homogenate, using a glass pestle to mix it in and grind up any lumps. Transfer the resulting wet granular mixture to a glass petri dish and spread it out to dry. As the mixture dries, transfer it back into the mortar, small portions at a time, and grind thoroughly until a fine, free-flowing powder is obtained.

2.3.3 Extraction

Pre-extract the soxhlet apparatus as described in Section 2.1.3, using a thimble of dimensions 43 mm I.D. by 123 mm external length and a 500 ml round bottomed flask. Soxhlet-extract the sample as described in Section 2.1.3, using 350 ml of 1:1 hexane:acetone.

2.4 Oil

A Florisil Sep-Pak is pre-eluted with 10 ml hexane and dried by pulling a vacuum through it. The Sep-Pak is placed in a 4 dram vial and tared. The oil is

thoroughly mixed and 200 mg is weighed directly into the Sep-Pak using a syringe. The Sep-Pak is then attached to a 10 ml glass syringe and the PCB's are eluted with hexane directly into a 10 ml volumetric flask. At this point the oil sample should be ready for GC analysis. If the extract is not colorless, a sulfuric acid wash may be necessary (see Section 3.4).

2.5 Air

2.5.1 Sample collection

Air samples are collected using a calibrated air sampling pump(Bendix Environmental and Process Instruments Division) equipped with a Florisil trapping tube(SKC Inc., Pa., cat # 226-39). The pump is placed near a contamination source, turned on, and allowed to run for 24 hours(note actual collection time). packing in the trapping tube is divided into The glass tube is broken and these sections. sections are poured into 2 dram vials and extracted separately in order to determine whether there is PCB breakthrough, which indicates sample overload.

2.5.2 Extraction

The Florisil fractions are shake-extracted 3 times for 20 minutes each time with small aliquots(approximately 1-2 ml) of hexane. The Florisil is allowed to settle after each extraction and the hexane is transferred to a 2 dram vial with a pasteur pipet. The combined hexane extracts are reduced in volume using a Pierce Reacti-therm Module(as described in Section 3.2) to 1.0

ml for analysis.

3.0 SAMPLE CLEANUP

3.1 Sodium sulfate drying

Add a sufficient quantity of anhydrous sodium sulfate to the crude extract such that the crystals are free-flowing. Pour a 10 cm column of sodium sulfate in a glass chromatography column, 2.5 cm I.D., equipped with a teflon stopcock. Wash the sodium sulfate with at least one bed volume of hexane. Pour the crude extract through the column(a funnel helps to load the column) and collect the eluate in an appropriately sized glass bottle. Rinse the sample flask, the funnel, the column walls and the sodium sulfate 3 times with small volumes of hexane and add the washings to the extract.

3.2 Reduction of sample volume

The sample volume is reduced to approximately 5 ml on a steam bath, using a Kuderna-danish equipped with a 3-ball Snyder column and a 10 ml concentrator tube. A pre-extracted boiling stone is added to prevent bumping. The extract is placed in the assembled apparatus, the sample bottle is rinsed 3 times with hexane, and the washings added to the apparatus. After the inner walls of the Kuderna-danish are rinsed down, the Snyder column is put in place, and a small volume of hexane is added at the top. The apparatus is placed in a steam bath and the solvent is evaporated. Take care to control the boiling intensity; solvent should not splash up high on the inner walls of the apparatus.

If a large volume must be reduced, add the extract in aliquots of approximately 200 ml, cooling before each addition, and adding a fresh boiling stone.

When the total extract volume is approximately 5 ml, cool. The extract is allow the apparatus to quantitatively transferred to a 4 dram vial to complete solvent evaporation. This is done as remove the Snyder column from the top of the apparatus. Carefully dry the joint connecting the concentrator tube and the Kuderna-danish so that condensation does not drip into the extract. Separate the pieces and transfer the extract to the vial using a pasteur pipet. Reconnect the pieces and wash the inside walls of Kuderna-danish with small volume a of hexane(approximately 2-3 ml). Add the washings to the extract and repeat the rinsing process 2 more times. The remainder of the solvent is evaporated under a stream of Femtogas nitrogen, using low heat(low setting 3.5 Pierce Reacti-therm Module). on Twenty-five microliters of hexadecane added is to prevent evaporative loss of lower chlorinated PCB isomers. When the sample reaches dryness, remove it from the heat source and reconstitute it in approximately 2-3 ml If there is a large amount of viscous or hexane. dark-colored residue a sulfuric acid wash may be necessary at this point(see Section 3.4). If not, Florisil chromatography is the next step.

3.3 Florisil chromatography

This procedure should result in a clear, colorless extract. The extract is passed through a Florisil Sep-Pak attached to a glass syringe and collected in a 10 m1 volumetric flask. Pre-elution of the Sep-Pak with 10 ml hexane is advisable. The 4 dram vial, then inner walls of the syringe are rinsed 3 times with hexane and the washings are passed through the Sep-Pak. The sample is adjusted to final volume with hexane and transferred to a screw-capped vial for storage. At this point the sample is ready for GC analysis. chromatogram shows the presence of sulfur or other extraneous peaks, a copper treatment for sulfur removal a concentrated sulfuric acid wash for hydrocarbon removal may be necessary.

3.4 Sulfuric acid wash

The sulfuric acid wash destroys hydrocarbons and colored biogenic compounds which appear as early-eluting peaks on the chromatogram. (If the presence of surfactants or alkali are suspected, water wash the extract before the sulfuric acid wash. water wash procedure is identical to the acid wash. not dry the resulting hexane solution with sodium sulfate before the acid wash, and reduce the volume to approximately 5 ml.) Add an equal volume of concentrated sulfuric acid to the hexane in a 4 dram The contents are mixed by inverting the vial several times (vigorous shaking can cause emulsions)

and the layers are allowed to separate. If an emulsion forms, sonicate the vial for several minutes. Do not add sodium chloride!

Remove the top hexane layer and wash the sulfuric acid layer two times with approximately 2 ml hexane. Combine all hexane solutions. Add a small amount of sodium sulfate to dry the extract, reduce the volume using the Reacti-therm module to 2-3 ml and pass the extract through a Florisil Sep-Pak as before.

(3.5 Copper treatment) *

This treatment removes sulfur compounds which may be found, especially in soil and sediment. The presence of sulfur is indicated as a large, early-eluting the baseline or as a large, offscale peak. The hexane extract is stirred with 0.5 grams of activated copper for 30 minutes.(copper activation: wash with 200 ml of 0.01M nitric acid, followed by successive 200 m1 rinses of distilled water. acetone. and hexane.Air-dry the powder, which should be red-orange and store in a screw-capped vial. should be used within 48 hr) The copper is allowed to settle and the hexane is removed. The vial and copper are rinsed 3 times with small amounts of hexane; the hexane solutions are combined. The solution is reduced in volume on the Reacti-therm and passed through a Florisil Sep-Pak.

Now use mencury instead, as in EPA Method 608

4.0 ANALYSIS

PCB's can be analyzed by either packed or capillary column GC, depending on the type of analysis required. Packed column analysis is routinely used to determine total PCB content for single Aroclors and mixtures. The weight percent of each peak in the chromatogram of single Aroclors has been determined by Webb and McCall Sci., 11,366(1973)). In addition, a (J. Chrom. program has been developed which calculates the total PCB's present, from monochloro- to octachlorobiphenyl, using a mixture of Aroclors 1242 and 1260, which spans range of isomers present in Aroclors the 1242,1248,1254, and 1260. For any mixture of 1242, 1254, and 1260 this program can also calculate the approximate percent composition, based on a peak ratio technique.

However, the isomer resolution afforded by packed column analysis is not sufficient to perform single isomer quantitation, the Aroclor composition of complex mixtures, or the relative percent contribution of each Aroclor when those percents are very different. Capillary GC is the preferred analysis when this type of information is required.

4.1 Packed column analysis

The instrument used for packed column analysis is a Hewlett Packard 5880 equipped with Autosampler model 7672A. The GC conditions are as follows:

Column=mixed silica, 1.5% SP2250 and 1.95% SP2401 on Supelcoport, 6 ft. by 0.25 in. O.D. glass

Oven temperature profile

equilibration time=1.0 min.

initial value=150 C

initial time=0.0 min.

level 1

program rate=2.0 C/min.

final value=210 C

final time=30.0 min

post value=150 C

nickel ECD detector temperature=300 C, signal B
injector temperature=300 C
injection volume=1 microliter
carrier gas

type=5% methane in argon
flow=60 ml/min.

chart speed=0.5 cm/min.

offset=10

attenuation=8

threshold=2

peak width=0.15

The sample is visually inspected for Aroclor type and the presence of interferences. Samples are then quantitated by comparing the chromatogram to that of a known Aroclor, using a multi-level external standard calibration based on peak height and covering the range of 0.1 to 10.0 PPM. Sample tracings of 10 PPM

standards of Aroclors 1242, 1254, and 1260 are shown in Figures 1, 2, and 3. Figure 4 shows a sample output of the type of information given in the program which calculates the total PCB content and relative percent composition.

4.2 Capillary column analysis

The instrument used for capillary column analysis is a Varian 4600 equipped with Autosampler model 8000 and a Vista 401 data system. The GC conditions are as follows:

column=DB-1 fused silica(J and W), 30 m.
oven temperature profile

initial value≈40 C

initial time=2.0 min.

level 1

program rate=10.0 C/min.

final value=80 C

final time=0.0 min.

level 2

program rate=6.0 C/min.

final value=225 C

final time=10.0 min.

nickel ECD detector temperature=300 C, signal A

range=10

make-up gas=nitrogen

injector temperature=300 C

injection volume=2 microliters

splitless injection

vent off at injection

vent on at 0.4 min. post injection

carrier gas type=helium

flow=30cm/sec.

chart speed

0.0-20.0 min=0.1 cm/min.

20.0-end=0.5 cm/min.

offset=10

attenuation=16

Samples are quantitated by comparison to a known Aroclor, using an external standard calibration based on peak height. Figures 5, 6, and 7 show capillary GC tracings of 2 PPM standards of Aroclors 1242, 1254, and 1260.

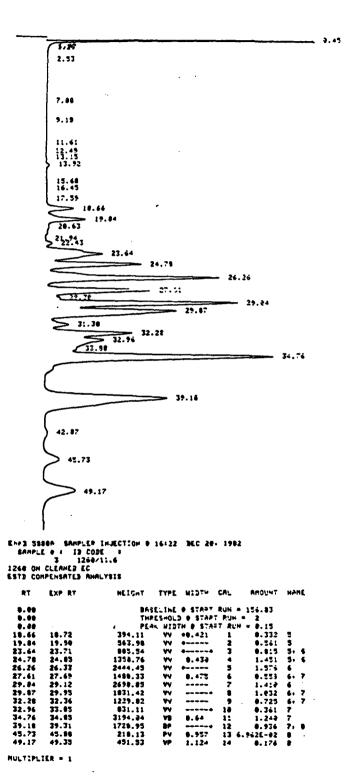
```
.... 1.45
                2.53
3.52
                 5.35
                               _ 7.97
                 8.3€
               Si.18.2
                                                                    _ 11.59
                           13.12.43
                  17.56
               19.16
                            20.02
              21,91
23.30
24.86
                22:3:
               > 27.59
               29.96
               31.96
32.81
33.63
               34.69
               32:22
               41.27
              55.30
56.20
               57.41
              58.77
            0V1 STOP RUN
EN-3 50000 SAMPLER INJECTION 0 20107 DEC 20- 1902
SAMPLE 0 1 ID CODE 1
3 1242/10.3
1260 ON CLEANED EC
ESTD COMPENSATED ANALYSIS
     PT
              EXP RT
                                     HEIGHT TYPE HISTH CAL
                                                                              ZMEM THUCKS
                                  9.00
5.50
0.00
3.52
5.53
6.17
7.07
9.16
10.51
11.59
12.43
14.01
15.69
16.41
18.24
20.02
22.48
23.30
24.86
27.59
             3.53
5.56
6.18
7.00
9.10
10.53
11.61
12.44
14.02
15.71
16.42
18.26
20.04
22.49
23.32
24.87
27.60
MULTIPLIER . 1
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-- 0.45
  2.52
   7.05
  8.34
9.15
9.73
        - 13.9e
    = 15.64
16.38
     1018.61
             19.84
     5 20.00
      21.89 22.41
                 23.24
    24.65
                       - 24.91
             - 26.22
              27.54
                 29.02
  29.67
30.92
31.91
    > 33.56
  5 24.60
  39.14
  42.79
  45.31
  47.23
  49.88
 33:73
---
```

E->3 58884 SAMPLEP INJECTION 0 00:34 JEC 21- 1982 SAMPLE 0 : 19 CODE : 7 1234/10.4 1260 OF CLERNED EC ESTB COMPENSATED ANALYSIS

RT	EXP RT	HEIGHT	TYPE	MIDTH	CAL	AMOUNT	NAME
		. 84	SELINE	. STAPT	RUN 4	164.37	
9.39		TH	RESHOL	D # STAR	T RU4	- 2	
0.30		PE	AK WID	TH . STA	RT RUN	= 0.15	
18.61	18.66	829.38	**		1	0.458	3
17.84	19.84	1236.43	W		2	1.195	5
23.24	23.64	1681.20	77		3	1.719	5. 4
24.91	24.70	1945.27	44	2.514	•	2.212	7: 6
26.22	26.26	1952.91	**	*	5	9.667	6
27.54	27.61	1377,48	W		4	9.524	6. 7
29.02	27.84	- 1764.33	77	0.475	7	0.724	6
29.87	29.87	267.61	w			9,129	6. 7
31.91	32.29	400.78	W		•	0.232	6. 7
33.56	32.96	351.48	w	*	10	0.154	7
34.68	34.76	279.68	w			.4516-42	7
39.14	39.18	102.10	BV	0.747		.3762-02	7. 8
45.51	45.73	3.97	82	*****		.201E-03	•
49.58	49.17	10,22	VB			.674E-03	i

MULTIPLIER = 1



```
13 R.R. 8638
1268 ON CLEANED EC
ESTD COMPENSATED AMALYSIS
                                                                                                                                                                                                                  0.45
   1.32
                                                                                                                                                                                                                                                                                                                                                                           HEIGHT TYPE MIDTH CAL
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 AMOUNT MARE
                                                                                                                                                                                                                                                                                                                                                                                          ### BASELIME ### STAPT RUM # 163.98

THATSHOLD ### STAPT RUM # 0.15

### STAPT RUM # 0.15

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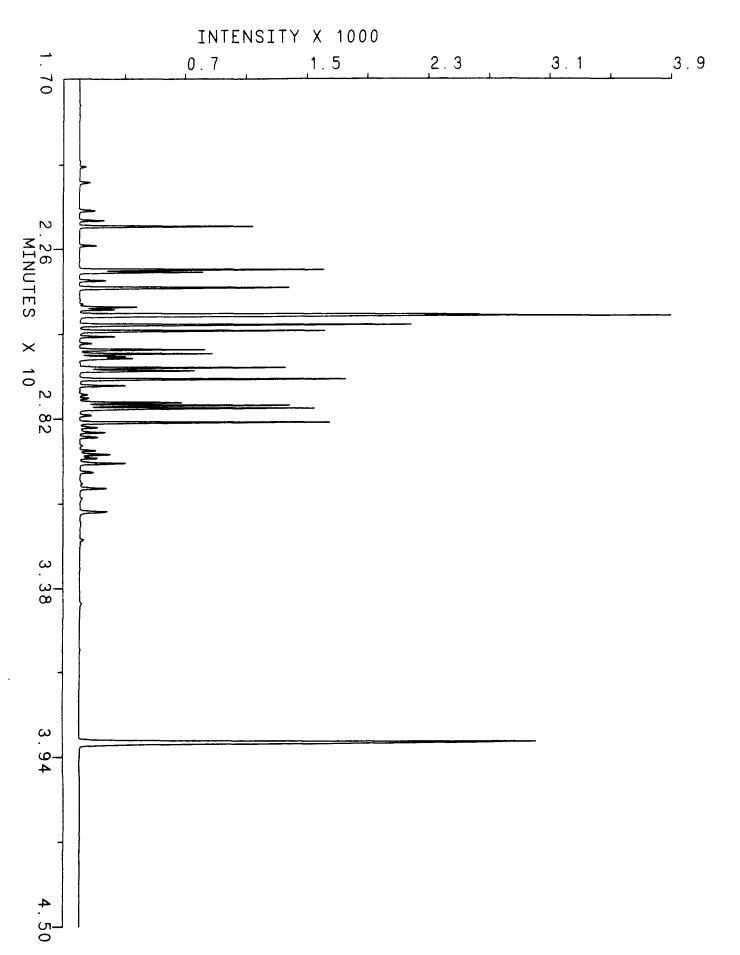
### STAPT RUM ###

### STAPT RUM ###
                                                                                                                                                                                                                                                                                                                                                                                             BASELINE # START RUN = 163.98
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9.15
11.56
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15.67
  1:13
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322.74
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   11,00
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5: 6
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23.32
24.86
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542.43
1841.50
    17.55
          > 18.62
         20.60 19.83
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                   → 27.24
                                                                                                                                                                                                                                                                                    RUN 40
                                         24.76
                                                                                                                                                                                                                                                                                    TOTAL BASED ON AROCHLOR 1242- 3.72321
                                                                                     - 26.23
                                                                                                                                                                                                                                                                                   PR: GETTIMG CALIS 1268 PEAKS T.P. FROM BEVICES 6
PP: DONE
END 3888A SAMPLER INJECTION 8 87:13 DEC 21, 1962
SAMPLE 8: 13 CODE:
13 R.R.9638
1268 ON CLEANED EC
ESTD COMPENSATED ANALYSIS
                                         27.58
                                                                    25.84
          31.24
         32.24
33.45
                                                                                                                                                                                                                                                                                               RT
                                                                                                                                                                                                                                                                                                                    EXP RT
                                                                                                                                                                                                                                                                                                                                                                           HEIGHT TYPE HIBTH CAL
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0.90
0.90
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THRESHOLD 0 START RUH = 2
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                                                                                                                                                                                                                                                                                       10.62
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23.50
24.76
26.23
27.58
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19.84
23.63
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0.483
0.523
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5, 6
5, 6
6, 7
6, 7
7, 7
                                                           39.:2
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27.61
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1650.52
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1.029
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29.81
29.84
32.24
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39.12
45.66
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525.41
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0.450
0.238
                                                                                                                                                                                                                                                                                                                     29.66
           45.66
                                                                                                                                                                                                                                                                                                                      32.95
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39.14
45.73
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12 6.558
13 4.289E-02
14 0.189
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                   49.45
                                                                                                                                                                                                                                                                                                                                                                                                                                1.123
                                                                                                                                                                                                                                                                                     MULTIPLIER . 1
                                                                                                                                                                                                                                                                                       TOTAL BASED ON AROCHLOR 1268- 7.25131
   54.76
                                                                                                                                                                                                                                                                                                                                                                     RHOUNT
       56.83
                                                                                                                                                                                                                                                                                                                                                              9.39921E-02
1.42006E-02
                                                                                                                                                                                                                                                                                                 HONDEWLDRD
                                                                                                                                                                                                                                                                                                 BICHLOPO
                                                                                                                                                                                                                                                                                                 TRICHLORD
TETRACHLORD
                                                                                                                                                                                                                                                                                                                                                               .101396
OV: STOP RUN
                                                                                                                                                                                                                                                                                                                                                             .094077
3.29923
2.40784
.429601
                                                                                                                                                                                                                                                                                                 PENTACHLORO
                                                                                                                                                                                                                                                                                    PENTACHLORO .574677
MEXACHLORO 3.29923
MEPTACHLORO 2.49784
OCTACHLORO .429681
TOTAL IF AROCHLOP 1242- 3.72321
TOTAL IF AROCHLOR 1268- 7.33131
TOTAL BASES ON MEDB & MCCALL = 8.88564
                                                                                                                                                                                                                                                                                                                                                                                                                                                       MICROCRAMS/ML
                                                                                                                                                                                                                                                                          PRI GETTING CALIB 1254 PEAKS T.P. FROM DEVICEO 6
PPI DONE

THE RHOD SEEEN SAMPLER INJECTION O 07:13 DEC 21. 1902
SAMPLE 0: 10 CODE:
17 R.R. 0638
1260 ON CLEANED EC
                                                                                                                                                                                                                                                                                      ESTD COMPENSATED AMALYSIS
                                                                                                                                                                                                                                                                                                                                                                           HEIGHT TYPE WISTH CAL
                                                                                                                                                                                                                                                                                               RT
                                                                                                                                                                                                                                                                                                                      EXP RT
                                                                                                                                                                                                                                                                                                                                                                                                                                                                               AMOUNT NAME
                                                                                                                                                                                                                                                                                        0.00
0.00
0.00
14.00
15.67
16.35
                                                                                                                                                                                                                                                                                                                                                                                              BASELINE O START RUN = 163.98
                                                                                                                                                                                                                                                                                                                                                                                            BASELINE 0 START RUN = 163.98
THRESHOLD 0 START RUN = 2
PERK MIDTH 0 START RUN = 0.15
20 8V ---- 2 5.7931-92
46 VV ---- 3 5.0251-92
74 VV ---- 5 0.365
75 VV ---- 7 0.123
43 VV ---- 8 0.413
36 VV 0-461 9 0.811
36 VV 0---- 18 1.630
                                                                                                                                                                                                                                                                                                                                                                       PE 247.32 89.00 82.46 322.74 501.56 152.17 542.43 1041.50 1586.34 842.59 1650.52 1186.52
                                                                                                                                                                                                                                                                                                                     13.90
15.65
16.40
19.62
19.04
22.42
23.24
                                                                                                                                                                                                                                                                                        19.62
19.83
22.38
                                                                                                                                                                                                                                                                                                                                                                                                              ******
                                                                                                                                                                                                                                                                                        23.26
24.76
26.23
                                                                                                                                                                                                                                                                                                                    24.82
26.23
27.54
29.83
29.87
31.89
                                                                                                                                                                                                                                                                                                                                                                                                                                                             10 1.630
11 8.164E-02
12 0.816
13 0.820
14 0.194
                                                                                                                                                                                                                                                                                                                                                                                                                          0.469
                                                                                                                                                                                                                                                                                        27.58
29.81
29.84
                                                                                                                                                                                                                                                                                                                                                                         1184.52
                                                                                                                                                                                                                                                                                          32.24
                                                                                                                                                                                                                                                                                     MULTIPLIER . 1
                                                                                                                                                                                                                                                                                     TOTAL BASED ON AROCHLOR 1254- 6.25165
                                                                                                                                                                                                                                                                                    PERCENT 1242- 3.29591
PERCENT 1254- 22.5638
PERCENT 1268- 74.1483
                                                                                                                                                                                                                                                                                                                                                                                                                  .266496
1.82442
3.99472
                                                                                                                                                                                                                                                                                                                                                                                                                                                     PPH 1242
PPH 1254
PPH 1260
```

7-23-87 GC1

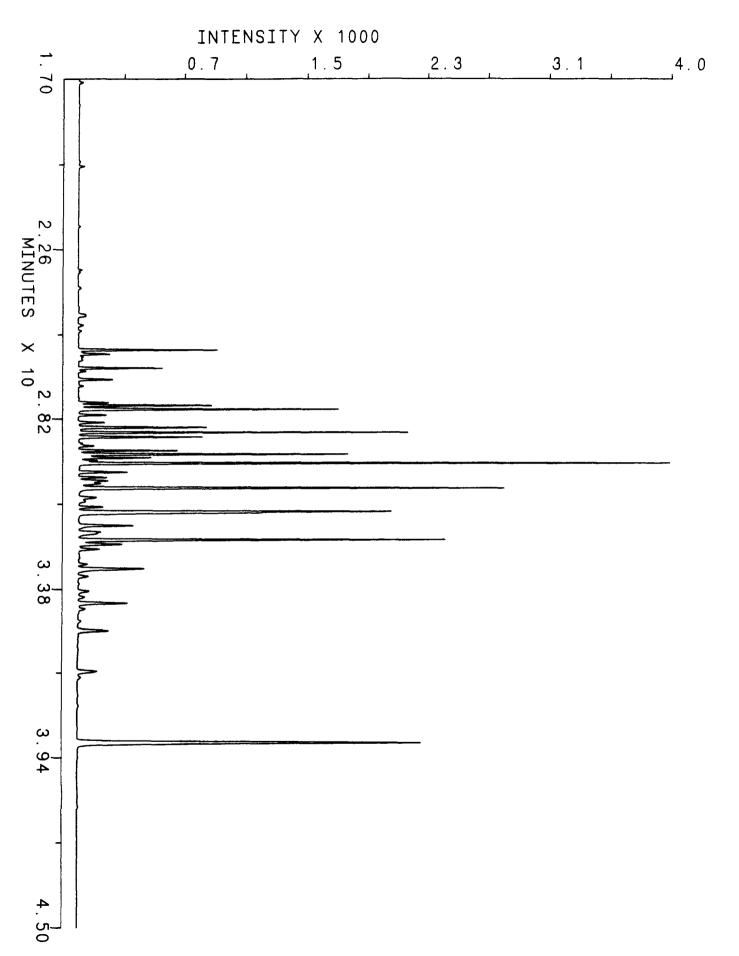
STDS004, 3, 1, A1242



6

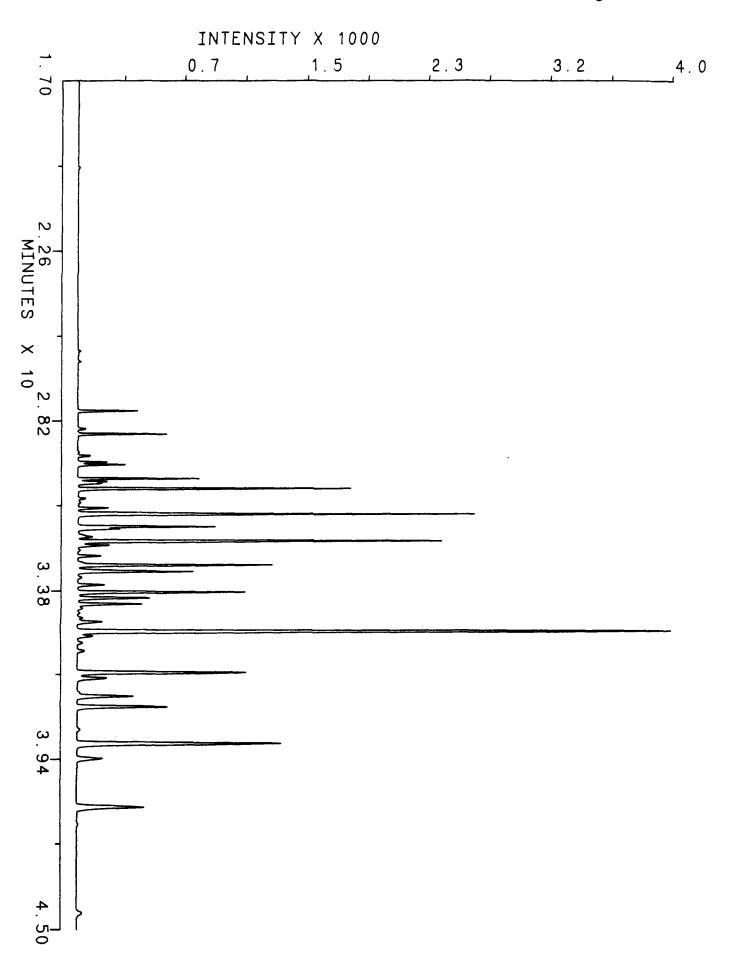
7-24-87 GC1

STDS006, 3, 1, A1254



7-24-87 GC1

STDS007, 3, 1, A1260



The ME	LROD EITS	18 MOLE.S	TUS			
RET. T	IME NAME	AREA	ART	RESPO	NSE PUNCTION	1
				R2*(AREA*2	+ RL AREA	+ R0
40.78	1	0.6	0.000000	R2 0.00000	R1 0.00000	7.00000
46.74	2	0.3	0.009683	0.00000	77.43000	0.00000
50.30 50.64	3	0.0	0.000000	0.00000	646.83002 131.64000	0.00000
52.36	3	3.8	0.040948	0.00000	25.85000	0.00000
54.80	6 7	5.7	0.007437	0.00000	3.13000	0.00000
55.63 56.12	'	7.3 45.7	0.024071 0.104588	0.00000	7.91000 5.49000	0.00000
57.46	j	0.0	0.000000	0.00000	7.22000	0.00000
57.73	10	5.0	0.010175	0.00000	8.72000	0.00000
58.89 59.18	11 12	0.4	0.000447 0.000000	0.00000	2.68000 48.99000	0.00000
59.48	13	0.6	0.002904	0.00000	11.61000	0.00000
59.79	14	67.7	0.210016	0.00000	7.47000	0.00000
59.99 60.71	15 16	35.0 8.8	0.094545 0.014857	0.00000	6.48000 4.05000	0.00000
61.33	17	73.5	0.106319	0.00000	3.47000	0.00000
62.08	18	0.2	0.000367	0.00000	4.40000	0.00000
62.31 62.72	19 20	0.9 1.3	0.001812 0.001880	0.00000	4.83000 3.47000	0.00000
62.95	21	15.#	0.023184	0.00000	3.52000	0.00000
63.16	22 23	9.9	0.015 97 1 0.140135	0.00000	3.87000 3.07000	0.00000
63.64 63.69	24	109.5 158.9	0.165599	0.00000	2.50000	0.00000
64.47	25	96.4	0.130201	0.00000	3.24000	0.00000
64. 98 65.44	26 27	69.3 10.8	0.059799 0.019629	0.00000	2.07000 4.36000	0.00000
65.78	28	0.0	0.000000	0.00000	7.46000	0.00000
66.06	29	3.8	0.012815	0.00000	8.09000	0.00000
66.37 66.57	30 31	0.0 33.4	0.000000 0.068224	0.00000	6.34000 4.90000	0.00000
66.93	32	35.1	0.053845	0.00000	3.68000	0.00000
67.19	33	13.5	0.020428	0.00000	3.63000	0.00000
67.32 67.51	34 35	16.7 0.0	0.029239	0.00000	4.20000 2.21000	0.00000
67.81	36	1.0	0.002447	0.00000	5.87000	0.00000
60.11	37	54.6	0.067599	0.00000	2.97000	0.00000
68.37 69.09	38 39	48.8 79.9	0.066501 0.0642 8 3	0.00000	3.91000 1.93000	0.00000
69.17	40	0.0	0.000000	0.00000	2.85000	0.00000
69.40	41 42	0.5 13.6	0.001065 0.018255	0.00000 0.000 00	5.11000 3.22000	0.00000 0.000 0 0
69.63 70.07	43	0.6	0.000913	0.00000	3.65000	8.00000
70.46	44	2.9	0.004485	0.00000	3.71000	
70.73 71.07	45 46	3.1 30.6	0.004252 0.031125	0.0000 0 0.00000	3.29000 2.44000	0.00000 0.00000
71.30	47	59.2	0.070580	8.00000	2.86000	0.00000
71.56	48	74.3	0.074954	0.0000	2.42000	0.00000
72.17 72.76	49 50	5.5 77.2	0.007681 0.091075	0.00000 0.00000	3.35000 2.83000	0.00000 0.00000
73.18	51	6.6	0.007263	0.0000	2.64000	0.0000
73.41	52	0.7	0.000832	0.00000	2.85000	0.0000
73.59 73.97	53 54	8.2 5.7	0.012 989 0.0065 8 2	0.000 0 0.00 00	3.80000 2.77 000	0.00000 0.00000
74.51	55	0.4	0.000397	0.00000	2.38000	9.00000
74.72	56 57	1.2	0.002266	0.00000	4.53000 2.32000	8.00000 8.00000
75.13 75.46	58	4.9 10.1	0.004739 0.006526	0.00000	1.55000	0.0000
75.77	59	6.5	0.004362	0.00000	1.61000	0.00000
76.03	60	0.0	0.00000	0.00000	3.58000	9.00000 9.00000
76.21 76.68	61 62	16.5 0.0	0.011280 0.000000	0.00000 0.00000	2.81000	0.00000
76.95	63	4.5	0.004014	0.00000	2.14000	0.00000
77.42	64 65	0.2 0.7	0.00025 8 0.000694	0.00000	3.10000 2.38000	0.00000 0.00000
77.72 77.79	66	0.0	0.000000	0.00000	1.89000	0.00000
77.93	67	1.2	0.001296	0.00000	2.59000	0.00000
78.15 78.34	69	0.0 10.3	0.000000 0.00 8 716	0.00000	2.41000 2.03000	0.00000 0.00000
78.45	70	0.0	0.00000	0.00000	2.29000	0.00000
79.19	71	1.4	0.001494 0.000231	0.00000	2.56000 1.85000	0.00000
79.34 79.91	72 73	0.3 0.1	0.000117	0.00000 0.00000	2.80000	0.00000
80.29	74	11.7	0.005316	0.00000	1.09000	0.00000
90.44 81.27	75 76	0.0 0.8	0.000000	0.0000 0	3.32000 1.43000	0.00000 0.00000
81.47	17	0.6	6.900280	0.00000	1.12000	0.00000
81.65	78	• .	0.900000 0.000190	0.00000	3.43000 2.2 8006	1.00000 1.00000
\$2.04 \$2.17	79 80	0.2 0.2	0.000228	0.0000	2.74900	1.0000
82.31	81	0.0	0.00000	0.00000	1.13000	0.0000
82.71 83.87	82 83	2.0 0.4	0.001359 0.000283	0.00 000	1.63000	0.00 00
83.45	64	4.2	0.000193	0.00000	2.31000	1.00000
83.93	85 86	0.0 0.0	0.000000	0.00000 0.00000	2.17000 1.15000	0.00000 0.00000
84.29 84.50	87	0.0	0.000000	0.00000	3.15000	0.0000
84.73	49	.0	0.00000	0.00000	1.88000	0.00000
85.15	!	0.7	0.000371 0.000000	0.00000 0.00000	1.27000	0.00000 0.00000
85.27 85.76	90 91	0.0 0.0	0.00000	8.00000	3.76000	9.00000
DG . 40	92		0.00000	0.00000	1.47000	0.00000
87.20 87.51	93 94	0.0	0.000181 0.000000	0.000 00 0.00000	1.45000 1.89000	4.00000 0.00000
88.13	95	1.4	0.000928	0.00000	1.59000	0.00000
88.38	96	0.0	0.00000	0.00000	1.03000	0.00000
88.49 86.87	97 98	0.0	0.000000 0.000000	0.00000 0.00000	1.00000 0.5 9 000	0.00000 0.00000
89.31	99	0.0	0.000000	0.00000	1.74000	0.00000
19.64	100	0.0	0.000000	0.00000	1.88000	0.0000
90.16 90.47	101 102	9.9 8.2	0.000000 0.000095	0.00 000 0.000 00	1.26000	0.00000 0.00000
90.85	103	0.0	0.000000	0.00000	0.85000	0.0000
91.39	104	0.0	9.000000	0.00000	0.81000	0.00000
92.14 93.93	105 106	9.8 9.0	0.000347 0.000000	0.00000	1.04000	0.00000 0.00000
94.40	107	0.0	0.000000	0.00000	1.40000	0.90000
95.63 95.96	108	8.e 0.6	000000.0 000000.0	0.00000 20000.0	1.12000	0.00000
96.36	110	0.0	0.000000	0.00000	1.22000	0.00000
96.77	111	0.0	0.00000	0.00000	0.80000	0.0000
100.00 101.23	I.S. 112	265.1 0.0	INT. STD. 0.000000	0.00000	1.00000 1.42000	0.00000
101.85	113	0.0	0.000000	0.00000	1.02000	0.0000
103.26	114	0.0	0.000000	0.00000	0.91000	0.00000
105.42 106.79	115 116	0.0 0.0	0.000000 0.000000	0.00000 0.00000	1.07000 0.85000	0.99000
114.24	117	0.0	0.00000	0.00000	2.97000	0.00000
122.90	118	0.0	0.00000	0.00000	1.19000	0.00000
			1 967443			

ANT TOTAL = 1.967442

RET. TIME NAME	AREA	ART	RESPO		
40.40			R2*(AREA*2 R2	R1	RO
40.69 1 46.74 2	2.2 0.0	0.000000 0.000000	0.00 00 0.0 000	0.00000 77.43000	0.0000
50.30 3 50.64 4	0.0	0.000000	0.0000	646.83002 131.64000	0.00000
52.36 5	0.0	0.00000	0.0000	25.85000	9.00000
55.60 7	0.0 0.1	0.00 000 0.000 48 7	0.00 0 00 0.00000	3.13000 7. 9 1000	0.00000
56.12 8 57.46 9	0.0	0.002703	0.00000	5.49000 7.22000	0.00000
57.76 10	0.1	0.000537	0.0000	8.72000	0.00000
58.78 11 59.18 12	0.0	0.000000	0.00000 0.00000	2.68000 48.99000	0.0000
59.48 13 59.82 14	0.0 1.3	0.000000 0.005976	0.00000	11.61000	0.00000
60.03 15	0.7	0.002792	0.0000	6.48000	0.00000
60.69 16 61.31 17	0.2 1.5	0.00049 8 0.003203	0.00000	4.05000 3.47000	0.00000 0.00000
62.13 18 62.42 19	0.0 0.2	0.000000	0.00000	4.40000	0.0000
62.73 20	0.0	0.00000	0.00000	3.47000	0.00000
62.98 21 63.16 22	0.5 0.0	0.0010 8 3 0.000 00 0	0.00000	3.52000 3.87000	0.00000
63.60 23 63.70 24	2.8 2.5	0.0052 9 0 0.003 846	0.00000	3.07000 2.50000	0.00000 0.00000
64.52 25	2.4	0.004786	0.00000	3.24000	0.00000
65.48 27	1.4	0.0017 83 0.001342	0.00000	2.07000 4.36000	0.00000
65.78 28 66.04 29	0.0	0.000000 0.000 9%	0.00000	7.46000 8.09000	0.00000
66.37 30 66.58 31	0.0 37.7	0.000000 0.113687	0.00000	6.34000 4.90000	0.00000
66.94 32	10.8	0.024459	0.00000	3.68000	0.00000
67.20 33 67.33 34	1.7	0.0037 98 0.002326	0.00000 0.00000	3.63000 4.20000	0.00000
67.46 35 67.78 36	1.6	0.002176 0.000000	0.00000	2.21000 5.87000	0.00000 0.00000
68.12 37	23.5 2.5	0.042953	0.0000	2.97000 3.91000	0.00000
69.10 39	11.5	0.013659	0.00000	1.93000	0.00000 0.00000
69.17 40 69.46 41	0.0 0.5	0.000000	0.00000	2.85000 5.11000	0.00000 6.00000
69.67 42 70.08 43	1.9	0.003765 0.000 899	0.00000	3.22900 3.65000	0.00000
70.46 44	0.2	0.000457	0.00000	3.71000	0.08000
70.75 45 71.08 46	9.1	0.001215 0.013665	0.00000	3.2 9000 2.44000	0.00000 8.00000
71.34 47 71.67 48	39.6 84.3	0.069700 0.125550	0.00 0 00 0.0000	2.86000 2.42 00 0	0.00000 0.000 00
72.16 49 72.78 50	9.4 9.0	0.01 9386 0.015675	0.00000 0.00000	3.35000 2.83000	0.000 08
73.19 51	42.5	0.069058	0.00000 0.00000	2.64000 2.85000	4.00000 4.00000
73.62 53	0.5 93.8	0.219361	0.0000	3.80000	8.80000
74.01 54 74.55 55	34.5 1.7	0.05 00 13 0.0024 90	0.00000 0.00000	2.77008 2.38000	0.80000 0.08600
74.76 56 75.17 57	5.3 29.7	0.014776 0.042405	0.00000	4.53000 2.32000	0.00000
75.48 58	82.4	0.078602	0.00000	1.55000	0.00000
75.78 59 76.02 60	24.3 5.9	0.024077 0.012999	0.0000 8 0.00000	1.61000 3.58000	0.00000
76.25 61 76.68 62	171.0 0.9	0.1725 89 0.001556	0.00000	1.64000 2.81000	0.00000 0.00000
76.99 63 77.43 64	14.7	0.019368	0.00000	2.14000 3.10000	9.00000
77.71 65	14.1	0.020652	0.00000	2.38000	0.00000
77.94 67	8.8	0.014027	0.0000	2.59000	0.0000
78.15 68 78.35 69	0.0 161.5	0.000000 0.201763	0.000 0 0 0.000 0	2.410 00 2. <i>0</i> 30 00	0.00000 0.00000
78.77 70 79.13 71	0.3 9.5	0.000423 0.014 96 7	0.00000	2.29066 2.56000	0.00 00 0.00 00
79.46 72 79.95 73	4.6	0.005237 0.015509	0.0000	1.85000	0.00000 0.00000
80.33 74	113.8	0.076338	0.00000	1.09000	0.00000 0.00000
80.44 75 81.26 76 81.52 77	53.1	0.108494	0.0000	3.32000 1.43000	0.0000
81.65 78	21.8 8.0	0.015026 0.000000	0.00006 0.00000	1.12000 3.43000	4.00000
82.88 79 82.19 88	8.9 7.8	0.0124 08 0.013153	0.0000 0.0000	2.20000 2.74000	1.0000
02.31 01 02.72 02	136.4	0.000000	0.00000	1.13000	0.00000
83.00 83	17.6	0.010413	0.00000	1.70000	0.0000
93.50 84 93.90 85	8.6 1.3	0.012226	0.0000	2.31006 2.17006	0.00000
84.29 86 84.50 87	0.0 0.0	0.000000 0.00000	0.0000	1.15000 3.15000	0.00 000
84.76 88 85.17 89	4.2 32.6	0.004859	0.00000	1.88000	0.0000
85.27 98	0.0 5.3	9.000000	0.00000 0.00000	1.60000	0.0000
95.81 91 86.43 92		0.012264 0.000724	0.00000	1.47000	0.00000
87.84 93 67.56 94	6.1 3.5	0.005443 0.004071	0.90 086 0.0000	1.45000	0.00000
88.07 95 88.30 96	23.2 0.0	0.0227 0 2 0.000000	0.00000 0.00000	1.59000 1.03000	0.00000 0.00000
88.56 97 88.87 98	4.2	0.002585 0.000218	0.00000	1.00000	0.00000 0.00000
89.31 99	0.6	0.00000	0.0000	1.74000	0.00000
89.72 100 96.16 101	1.0 6.0	0.001157 0.00000	0.00000 0.00000	1.88000 1.26 <i>000</i>	0.00000 00000.0
90.51 102 91.00 103	15.6 0.2	0.010945 0.000105	0.00000 0.00000	1.14000 0.85000	0.00008
91.44 104 92.13 105	0.6	0.000299	0.00000	0.81000 1.04000	0.0000
93.98 106	11.6	0.000138	0.00000	1.14000	0.00000
94.47 107 95.68 108	2.4 0.3	0.002068 0.000207	0.00000 0.00000	1.40006	0.0000
96.02 109 96.86 110	0.9 1.0	0.000847 0.000751	0.00000 0.00000	1.53000 1.22000	0.00 00 0.00 000
98.84 111 100.08 I.E.	0.3 190.5	0.000148 INT.STD.	0.00000	0.80000 1.00000	0.0000
101.34 112	0.4	6.000350	0.00000	1.42000	0.00000
101.85 113 103.26 114	0.0	0.000000	0.00000	0.91000	0.00000
105.50 115 106.79 116	0.8	G.000527 G.000000	0.00000 0.00000	1.07000 0.85000	0.00000
114.24 117 122.90 118	0.0	0.000000	0.0000	2.97000 1.19000	0.00000
	····				

ANT TOTAL - 2.003465

STDS007,3,1,A1260 The METHOD file is MOLE.STDS

The HETHO	D file is	MOLE.STD	s			
RET. TIME	NAME	AREA	ART	RESPON R2* (AREA^2)		RC
40.69	1	1.1	0.00000	R2 0.00000	R1 0.00000	RG 0.00000
46.74	Ž	0.0	0.00000	0.00000	77.43000	0.00000
	3 4		0.00 000 0.00 00 0		646.83002 131.64000	0.00000
52.36	5 6	0.0	0.00000	0.00000	25.85000	0.00000
55.63	7	0.0	0.00000	0.00000	3.13000 7.91000	0.00000
	t ,		0.000000 0.00000	0.00000 0.00000	5.49000 7.22000	0.00000
57.73	10	0.0	0.00000	0.00000	8.72000	0.00000
	11 12		0.00000 0.00000	0.00000 0.00000	2.68000 48.99000	0.00000
59.48	13 14		0.00000 0.00000	0.00000	11.61000	0.00000
59.99	15	0.0	0.00000	0.00000	6.48000	0.00000
	1 6 17		0.000000 0.000000	0.00000	4.05000 3.47000	0.00000
62.13	18	0.0	0.00000	0.0000	4.40000	0.00000
	19 20		0.00000	0.00000 0.00000	4.83000 3.47000	0.00000
	21 22		0.000 00	0.00000 0.00000	3.52000 3.87000	0.00000
63.64	23	0.0	0.00000	0.00000	3.07000	0.00000
	24 25		0.000000 0.000000	0.0000	2.50000 3.24000	0.00000
	2 6 27		0.000300 0.000000	0.00000	2.07000 4.36000	0.00000 0.00000
65.78	28	0.0	0.00000	0.00000	7.46000	0.00000
66.37	29 30	0.0	0.000000	0.00000	#.09000 6.34000	0.00000
	31 32).004267).000267	0.00000	4.90000	0.00000
67.19	33	0.0	0.00000	0.0000	3.63000	0.00000
67.43	34 35	1.2	0.000000 0.001925	0.00000 0.00000	4.20000 2.21000	0.00000
	36 37	0.0	0.000000 0.000 86 2	0.00000 0.00000	5.87000 2.97000	0.00000
68.37	38	0.0	0.000000	0.00000	3.91000	0.00000
	39 40		0.000420 0.000000	0.00000 00000.0	1.93000 2.85000	0.00000
69.41	41 42	0.2	0.000742	0.00000 0.00000	5.11000 3.22000	0.00000
70.07	43	0.0	.000000	0.0000	3.65000	0.00000
	14 15	0.0	0.00000	0.00000 0.00000	3.71000 3.29000	0.0000
71.05	16 17	0.2	.000354	0.00000	2.44000 2.86000	0.00000
71.65	48 :	17.6 (.030910	0.00000	2.42060	0.00000
	49 50		.0004 86 .0002 0 5	0.00000	3.35000 2.83000	0.00000
73.16	51 52	3.2	.006131	0.0000	2.64000 2.85000	0.00000
73.60	53 :	24.1 0	.066462	0.00000	3.80000	0.00000
	54 55	0.6	.001206	0.00000 0.00000	2.77000 2.3 800 0	0.000 00
74.73	3 6 37	0.0	.000 000	0.0000	4.53000 2.32000	0.00000
75.45	58	4.5	.005062	0.00000	1.55000	0.0000
	59 50		0.00 046 7 0.0241 6 2	0.00000	1.61000 3.58000	0.00000
76.20	1	14.0	.016663	0.0000	1.64000	0.00000
76.94 6	i2 i3	0.2	.00 0000	0.0000	2.14000	0.00000
	i4 : i5		.075142 .015 02 7	0.0000 0.0000	3.10000 2.3 8 000	0.00000
77.79 6	56	1.3 0	.011384	0.00000	1.89000	0.0000
78.15 6	i7 i8	0.0	.000000	0.00000	2.41000	0.0000
	59 (70	97.6 0	.129055 .000000	0.00 00 9.00 00	2.03000 2.2 900 0	0.00000
79.10 7	11	3.1 0	.005759	0.88000	2.56000 1.85000	0.00000
79.90 7	73 1	10.7 0	.021743	0.00000	2.80000	0.00000
	74 75 1!	0.0 0 34.3 0	.000000 .371773	0.0000	1.09000 3.32000	0.00000
81.25 7	16 17	0.0	.038365	0.0000	1.43000 1.12000	0.00000
81.65 7	18]	14.2 0	.035347	0.0000	3.43000	0.0000
	19 16	0.0	. 000000	0.00000	2.74000	0.00000
)1 2 1!	8.8 G	.007217 .178269	0.00000	1.13000 1.63000	0.0000
83.63	13 1	2.2 0	.015052	0.00000	1.70000	0.0000
	14 15	9.3	.002850 .014646	0.00000	2.31000 2.17000	0.00000
84.22	1 6 17	0.1	.000083 .005029	0.00000	1.15000 3.15000	0.00000
84.73 8	18 1	72.2 0	.096567	0.99000	1.88000	0.00000
	19 10 (0.0 0 18.4 0	.000 000	0.00000 0.00000	1.60000	0.00000
85.76 9	2	2.6	.007095	0.00000		0.00000 0.00000
96.99 9	13 (9.4 0	.073030	0.00000	1.45000	0.00000
	14 3 15 2	2.4 0 6.8 0	.044441 .030925	0.00000	1.59000	0. 00000 0. 00000
80.38 9 88.49 9	16 17	3.3 0	.002467 .000 00 0	0.00000		0.00000 0.00000
88.87 9	•	1.8 0	.000771	0.00006	0.59000	0.0000
) 9 .00 1		.003662 .016372	0.00000 0.00000	1.88000	0.00000 0.00000
90.16 1	.01	0.6 0	.000000	0.88088	1.26000	0.0000
90.85 1	.03	8.5 0	.005243	0.00000	0.85000	0.0000
92.06 1	04 05	4.6 0	.001999 .003472	0.0000	1.04000	0 . 00000 0 . 00000
93.93 1	06 8	8.6 0	.073361 .017679	0.00000	1.14000	0.00080
95.63 1	08	0.0 0	.000000	9.00000	1.12000	0.00000
96.86 1		2.0 0 0.3 0	.035532 .044535	0.00000 0.00000	1.22008	0.00 000 0.00000
96. 77 1	11	2.7 0	.001568	0.00000	8.80000	0.00000
101.23 1	12 1	6.9 0	WT.STD. .017416	0.00000	1.42000	0.00000
			.000 666 .00052 8	0.00000 0.00000	9.91000	00000.0 00000.0
105.42 1	15 4	5.3 0	.035177 .001110	0.00000	1.67000	0.00000 0.00000
114.24 1	17	6.5 0	.014010	0.0000	2.97000	0.0000
122.90 1	18	0.0 0	.000000	0.0000	1.19000	0.00000

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